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Selective Flocculation and Flotation of a Mesabi Range Hematitic-Goethitic Taconite

By H. D. Jacobs and A. F. Colombo

UNITED STATES DEPARTMENT OF THE INTERIOR

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SELECTIVE FLOCCULATION AND FLOTATION OF A MESABI RANGE HEMATITIC-GOETHITIC TACONITE

by

H. D. Jacobs[1] and A. F. Colombo[2]

ABSTRACT

The Bureau of Mines undertook a research program to insure an adequate supply of iron raw materials for future iron and steel making needs. A sample was obtained of oxidized taconite from the western Mesabi Range in Minnesota, and the metallurgical response of the taconite to beneficiation by a process that included selective flocculation-desliming-cationic flotation was evaluated. The sample contained 35.3 pct iron and represented approximately 2 billion tons of crude material. Selective flocculation and flotation were evaluated in a pilot plant with a feed rate of 900 lb/hr. Concentrates averaging 61.7 pct iron and 5.1 pct silica were obtained, with an accompanying iron recovery of 67.2 pct. About 83 pct of the process water requirements were reclaimed from the slime and froth tailing and then recycled.

INTRODUCTION

The United States imported approximately 29 pct of its ore requirements for iron and steel making in 1978 (8, 11)[3] and will require increased tonnages of imports as the steel industry expands to meet future needs. Most of the domestic raw materials used in 1978 came from pelletized magnetite concentrates. However, as the demand for iron-bearing raw materials increases, western Mesabi Range oxidized (nonmagnetic) taconites,[4] which are a major domestic resource, containing an estimated 10 billion tons[5] of material assaying 30 to 40 pct iron, will have to be used. The oxidized taconites cannot be beneficiated by the wet low-intensity magnetic separation method used to

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[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.
[4]The term "oxidized taconite," synonymous with "nonmagnetic taconite," refers to the iron oxides that are not recovered by a drum-type magnetic separator having a maximum field intensity of 3,000 gauss at the drum surface.
[5]Throughout this report, ton refers to long ton, or 2,240 pounds.

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upgrade magnetite taconites. Therefore, alternative beneficiation processes for treating the oxidized taconites are being evaluated at the Twin Cities (Minn.) Research Center of the Bureau of Mines to provide the technology to insure an adequate future supply of iron raw materials for the iron and steel industry.

In 1973, the Bureau began a comprehensive program on the beneficiation of western Mesabi Range oxidized taconites. The objectives of the investigation were (1) to increase availability of domestic iron ores through demonstrated amenability of oxidized taconites to beneficiation, (2) to make an economic and metallurgical comparison of the alternative processes: selective flocculation-flotation and reduction roasting-magnetic separation-flotation, and (3) to develop new and improved beneficiation methods for oxidized taconites. The iron potential of the western Mesabi Range oxidized taconites was to be evaluated by taking three bulk samples, containing 350, 600, and 800 tons, respectively, with different mineralogical makeups and representing large blocks of material. The metallurgical responses of these samples to the two alternative processes previously mentioned were evaluated. Both processes have been investigated (1-6, 9-10, 12-13), but require further development to reduce processing costs and to establish that they are applicable to the range of ore types likely to be encountered. This report summarizes the data obtained from the application of the Bureau-developed selective flocculation and flotation process (7) to the first of the three bulk samples.

SAMPLE DESCRIPTION

A 350-ton sample, which represents about 2 billion tons of oxidized taconite, was obtained from the iron formation located near Nashwauk, Minn., and crushed to a nominal minus 5/8-inch size. Table 1 shows the mineral distribution in the bulk sample. The iron oxides in the sample were primarily hematite and goethite; hematite-to-goethite ratio was 1.1:1. Most of the hematite and magnetite grains were from 30 to 300 μm and averaged about 100 μm. The goethite particles ranged from a few micrometers to 1.5 mm and averaged approximately 40 μm. The hematite pseudomorphically replaced magnetite, thus preserving the original shape of the octahedral magnetite, which usually occurred in clusters with granule shapes and occasionally occurred as single grains. A small amount of hematite replaced siderite and iron silicates; occasionally, goethite replaced bladed silicates. Most of the goethite was present as irregular shapes because it filled cavities where quartz or chert was leached out. Much of the goethite was also coarser than the hematite and magnetite and had no apparent crystal outline or habit. The magnetite occurred as unaltered grains and unoxidized cores in hematite particles. Liberation of the iron oxides from the gangue minerals was between 20 and 50 μm. Table 2 shows the average chemical analysis of the taconite, and an average screen analysis of the crude taconite is given in table 3.

TABLE 1. - Mineral distribution in bulk sample

Mineral	Wt-pct
Quartz	44.8
Hematite	25.9
Goethite	22.8
Magnetite	4.1
Siderite	2.1
Iron silicates	.3
Total	100.0

TABLE 2. - Chemical analysis of crude taconite, wt-pct

Fe	35.3	CaO	0.52	Na_2O	0.03
Fe^{2+}	2.1	S	.018	C	.17
SiO_2	43.7	P	.039	TiO_2	.25
Al_2O_3	.65	Mn	.47	LOI at 400 ° C	2.26
MgO	.19	K_2O	.03	LOI at 1,000° C	3.17

LOI Loss on ignition.

TABLE 3. - Screen analysis of crude taconite, pct[1]

Size fraction[2]	Weight	Iron	Iron distribution
Plus 1/2 inch	9.9	36.9	10.3
Minus 1/2 plus 3/8	22.0	36.8	22.9
Minus 3/8 plus 1/4	12.4	39.5	13.9
Minus 1/4 inch plus 4 mesh	8.8	39.0	9.7
Minus 4 plus 10	12.8	39.0	14.1
Minus 10 plus 28	6.8	40.7	7.8
Minus 28 plus 65	7.6	40.4	8.7
Minus 65 plus 100	1.4	34.0	1.4
Minus 100 plus 200	5.5	28.0	4.4
Minus 200 plus 325	4.2	20.2	2.4
Minus 325 plus 500	3.5	15.1	1.5
Minus 500 mesh	5.1	19.7	2.9
Composite	100.0	35.3	100.0

[1]Rod mill feed.
[2]Tyler Standard sieve size.

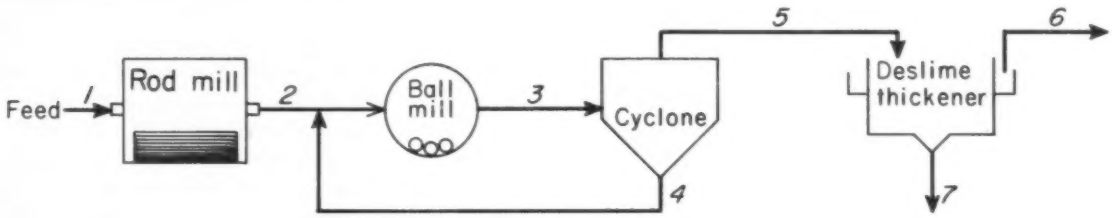
PILOT PLANT RESEARCH

Methods for upgrading the fine-grained oxidized taconites found in the Michigan and Minnesota iron ranges include the selective flocculation-gangue flotation system using either an anionic or a cationic collector. After extensive research and development on the two flotation systems, the cationic flotation process was determined to be the simplest system to apply commercially. The selective flocculation-cationic flotation system includes (1) stage grinding of the oxidized taconite, with dispersants to liberate the iron

minerals from the gangue, (2) selective flocculation of the iron minerals with starch, (3) rejection of a siliceous slime, and (4) finally, cationic flotation of the selectively flocculated material to yield a froth tailing and an iron oxide concentrate.

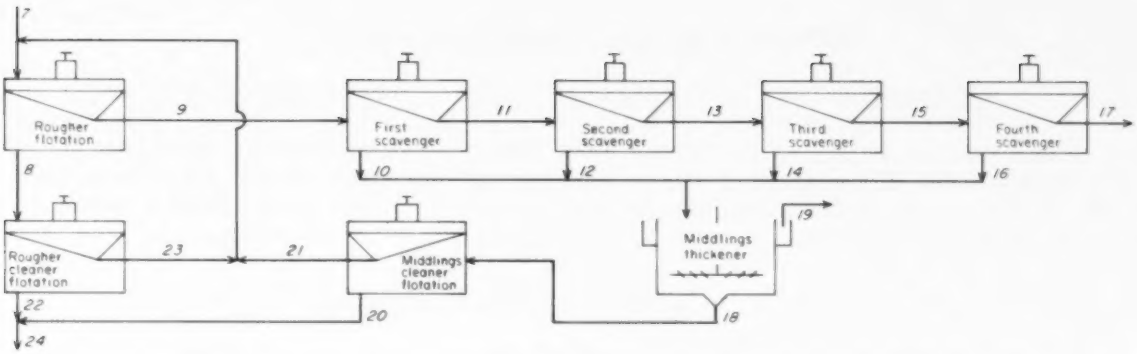
Flowsheet, Equipment, and Test Conditions

Figures 1 and 2 show the selective flocculation and flotation process and include the metallurgical data obtained during the operation of the pilot plant. Tables 4 and 5 show the equipment used in the pilot plant research. Minus 5/8-inch oxidized taconite at 900 lb/hr was diluted to 70 pct solids with reclaimed water and fed to a rodmill containing a seasoned charge of hot-rolled carbon steel rods (table 6). Sodium hydroxide and sodium silicate were added to the rodmill to disperse the pulp and to regulate the pH at 11. A rod wear rate of 2.1 lb/ton was obtained during grinding to 42 pct minus 400 mesh. The average screen analysis of the rodmill discharge and other products in the grinding circuit are reported in table 7. Rodmill discharge was pumped to an overflow-type ball mill, which ground the taconite to 58 pct minus 400 mesh. The seasoned charge of balls shown in table 6 had a wear rate of 4.4 lb/ton. Ball mill discharge was diluted to 42 pct solids with reclaimed water and then sized in a hydrocyclone. Cyclone underflow, averaging 488 wt-pct of the plant feed, was returned to the ball mill for regrinding. Cyclone overflow, averaging 94 pct minus 400 mesh, was conditioned with starch to



Analysis, pct	Stream number						
	1	2	3	4	5	6	7
	Stream name						
	Feed	Rod mill discharge	Ball mill discharge	Cyclone underflow	Cyclone overflow	Slime	Flotation feed
Iron	35.3	35.3	47.9	50.5	35.3	20.1	36.5
Weight	100.0	100.0	588.0	488.0	100.0	8.7	91.3
Iron distribution	100.0	100.0	798.0	698.0	100.0	5.0	95.0
Solids	94.8	70.0	67.1	65.0	16.5	1.7	33.8

FIGURE 1. - Grinding, selective flocculation, and desliming flowsheet.



Pct	Stream number										
	7	8	9	10	11	12	13	14	15	16	17
	Stream name										
	Flotation feed	Rougher flotation		First scavenger		Second scavenger		Third scavenger		Fourth scavenger	
		Underflow	Froth	Underflow	Froth	Underflow	Froth	Underflow	Froth	Underflow	Tailing
Iron	36.5	57.7	32.8	58.0	26.6	56.4	22.1	55.3	19.8	47.0	18.6
Weight	91.3	88.2	84.5	16.7	67.8	8.9	58.9	3.8	55.1	2.3	52.8
Iron distribution	95.0	144.2	78.5	27.4	51.1	14.2	36.9	6.0	30.9	3.1	27.8
Solids	33.8	15.4	19.4	11.0	20.0	6.7	17.8	4.6	15.5	5.0	14.1

Pct	18	19	20	21	22	23	24
	Middlings thickener		Middlings cleaner flotation		Rougher cleaner flotation		Combined concentrate
	Underflow	Overflow	Underflow	Froth	Underflow	Froth	
Iron	56.4	Neg	61.1	50.6	62.2	56.3	61.7
Weight	31.7	Neg	17.5	14.2	21.0	67.2	38.5
Iron distribution	50.7	Neg	30.2	20.5	37.0	107.2	67.2
Solids	28.3	Neg	29.5	11.0	23.5	11.3	26.1

[1] Neg. negligible.

FIGURE 2. - Rougher, scavenger, and cleaner flotation flowsheet.

selectively flocculate the iron oxides and then fed into a thickener. A slime product was separated at a rise rate of 1.6 in/min and was routed to the water treatment circuit. The thickener underflow was routed to the rougher flotation cells where Arosurf MG-98A,[6] a cationic collector, was added to the flotation feed box. Froth from the rougher flotation step was scavenged in four scavenger flotation steps. Starch was added to the rougher flotation froth to depress the iron oxides. The four scavenger underflows or middlings were thickened and then cleaned in a single flotation cell. A small amount of MG-98A collector was added to the middlings cleaner flotation step. The middlings thickener, which overflowed at a rate of about 6.7 gpm, was used to dilute the cyclone feed. The rougher concentrate was reagentized with a collector-froth mixture with a collector-to-frother ratio of 100:1 and up-graded in the rougher cleaner flotation cells. The underflows from the rougher and middlings cleaner were combined and became the final concentrate. The cleaner froths were returned to the rougher flotation step.

[6]Reference to specific brand names is made for identification only and does not imply endorsement by the Bureau of Mines.

TABLE 4. - Equipment shown in figure 1

Equipment	Size	Operating data
Rod mill	2 by 4 ft	Power consumption was 10.5 kwhr/ton.
Ball mill	3 by 3 ft	Power consumption was 19.4 kwhr/ton.
Cyclone	3 in	
Deslime thickener	3 by 3 ft	

TABLE 5. - Equipment shown in figure 2

Equipment	Size
Rougher flotation	Bank of 3 cells, 10 ft^3.
1st scavenger	Bank of 4 cells, 6 ft^3.
2d scavenger	Bank of 4 cells, 6 ft^3.
3d scavenger	Bank of 3 cells, 4.5 ft^3.
4th scavenger	Bank of 2 cells, 3 ft^3.
Middlings thickener	5 by 3 ft.
Middlings cleaner flotation	Bank of 1 cell, 1.5 ft^3.
Rougher cleaner flotation	Bank of 2 cells, 3.0 ft^3.

TABLE 6. - Rod and ball mill media charge

Rod mill				Ball mill		
Rod diameter, inches	Rods	Weight, lb	Weight, pct	Ball diameter, inches	Weight, lb	Weight, pct
1	14	40	2.6	5/8 by 3/4	440	20.0
1-1/2	15	340	22.4	3/4 by 7/8	990	45.0
2	19	760	50.2	7/8 by 1	660	30.0
2-1/2	6	375	24.8	1 by 1-1/4	110	5.0
Total	54	1,515	100.0	Total	2,200	100.0

TABLE 7. - Average screen analyses of grinding circuit products

Sieve size, mesh[1]	Cumulative wt-pct retained			
	Rod mill discharge	Ball mill discharge	Cyclone underflow	Cyclone overflow
Minus 28 plus 35	1.0	NAp	NAp	NAp
Minus 35 plus 48	2.6	NAp	NAp	NAp
Minus 48 plus 65	6.6	NAp	NAp	NAp
Minus 65 plus 100	13.6	NAp	0.6	NAp
Minus 100 plus 150	22.1	1.8	2.4	NAp
Minus 150 plus 200	31.6	4.6	7.1	NAp
Minus 200 plus 270	40.8	10.8	16.8	0.4
Minus 270 plus 325	49.4	24.4	36.2	1.2
Minus 325 plus 400	55.8	37.0	50.3	3.1
Minus 400	58.0	42.1	57.2	6.0

NAp Not applicable.

[1]Tyler Standard sieve size.

Metallurgical Results

The metallurgical results shown in table 8 are averaged analyses of the concentrate, slime, and tailing obtained from 40 consecutive hours of pilot plant operation. The average reagent suite utilized is given in table 9. Samples of the pilot plant streams were taken when equilibrium was achieved, or about 4 hours into the shift; five sample cuts were taken during a 1-hour period. The concentrate had an average grade of 61.7 pct iron and 5.1 pct silica with recovery of 67.2 pct of the iron in the feed. Table 10 is an average chemical analysis of the concentrates obtained. The slimes analyzed 20.1 pct iron and contained 5.0 pct of the iron in the feed. Table 11 shows the results obtained from using two types of starches in the selective flocculation step. Causticized pearl starch and C-165 pearl starch, which is similar to a jet-cooked pearl starch, gave similar results. The tailing averaged 18.6 pct iron and contained 27.8 pct of the iron in the feed.

TABLE 8. - Metallurgical results, pct

Product[1]	Weight	Iron	Silica	Iron distribution
Feed	100.0	35.3	43.7	100.0
Concentrate	38.5	61.7	4.1	67.2
Slime	8.7	20.1	NAp	5.0
Tailing	52.8	18.6	NAp	27.8

NAp Not applicable.
[1]Average of 5 tests over 40 consecutive hours.

TABLE 9. - Reagent suite

Reagent	Addition point	Addition, lb/ton	Function
Sodium hydroxide	Rod mill	2.82	Dispersant and pH regulator.
Sodium silicate	do	1.68	Dispersant.
C-165 starch or pearl starch.	Conditioner before deslime.	.56	Flocculant.
Do	Rougher flotation froth.	.56	Depressant.
Arosurf MG-98A	Rougher flotation feed.	.34	Collector.
Arosurf MG-98A and frother.[1]	Rougher cleaner flotation feed.	.14	Collector and frother.
Arosurf MG-98A	Middlings cleaner flotation feed.	.08	Collector.

[1]Mixture of 100 parts Arosurf MG-98A and 1 part Dowfroth 250.

TABLE 10. - Concentrate analysis, wt-pct

Fe	61.7	CaO	0.54	Na_2O	0.015
Fe^{2+}	3.7	S	.011	C	.2
SiO_2	5.1	P	.061	TiO_2	.29
Al_2O_3	.6	Mn	.40	LOI at 400° C	4.5
MgO	.21	K_2O	.013	LOI at 1,000° C	5.4

LOI Loss on ignition.

TABLE 11. - Effect of starch type on slime

Reagent[1]	Test period, hours[2]	Analysis, percent		
		Iron	Weight	Iron distribution
Pearl starch.....	16	20.2	10.1	5.8
C-165 pearl starch.....	24	20.0	7.4	4.2

[1]Constant addition of 0.56 lb/ton.
[2]Number of hours that reagent was used in the 40-hour test period.

Water Reclamation

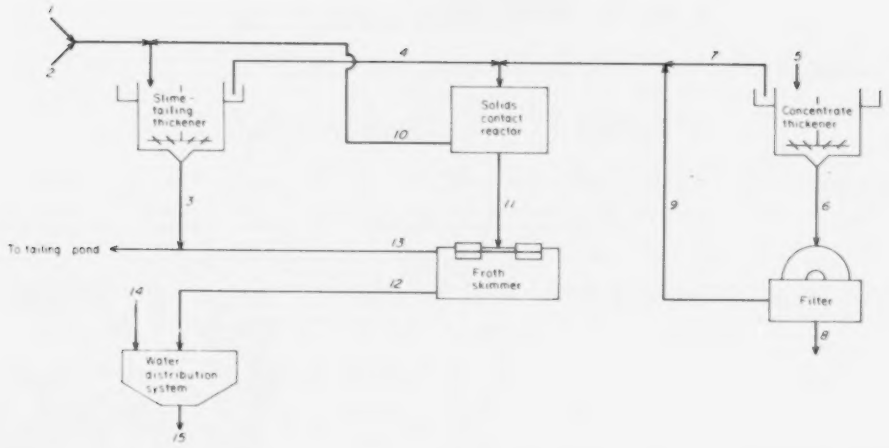
Figure 3 shows the water reclamation flowsheet used to treat the pilot plant waste streams, and the water reclamation equipment that was used is shown in table 12. Slime and froth tailing were combined and gave a 7.0 pct solids slurry, which was flocculated with 1.68 pounds of lime/ton and 0.035 pound of a cationic flocculant Superfloc 330 per ton and then fed to a thickener. The thickener overflow was pumped to a solids contact reactor (SCR), which is a rectangular, compartmented water treatment tank that employs the principles of sludge blanket clarification. The SCR underflow at 7.0 pct solids was recycled to the slime-tailing thickener, and the overflow was pumped to a froth skimmer to remove excess froth, which was pumped to the tailing pond. Slime-tailing thickener underflow, which was controlled at 50 pct solids to prevent excess water loss, was pumped to a tailing pond.

TABLE 12. - Equipment shown in figure 3

	Size
Slime-tailing thickener.....	6 by 6 ft.
Solids contact reactor.....	5 ft 8 in by 10 ft 7 in.
Concentrate thickener.....	5 by 5 ft.
Filter.....	3 by 1 ft.
Froth skimmer.....	Bank of 2 cells, 5.2 ft^3.
Water distribution system.....	Water supply tank, 160 gal.

Iron oxide concentrate was pumped to a thickener and flocculated with 0.035 pound of Superfloc 330 per ton. The thickener underflow was controlled at 60 pct solids and filtered on a rotary drum filter. Filter cakes with an average moisture of 12.5 pct and Blaine surface area of 2,855 cm^2/g were obtained. The filtrate was combined with the concentrate thickener overflow and pumped to the SCR.

The froth skimmer underflow was pumped to a water distribution tank, where makeup water was added to satisfy the requirements of the plant. An average of 83 pct of the total water used, or 13.9 gpm, was recovered and recylcled. The reclaimed water had an average turbidity of 1,360 ppm silica equivalent, pH of 10.8, and calcium ion content of 6.7 ppm.



Analysis	Stream number														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
	Stream name														
	Slime	Tailing	Slime-tailing thickener underflow	Slime-tailing thickener overflow	Concentrate	Concentrate thickener underflow	Concentrate thickener overflow	Filter cake	Filtrate	Solids contact reactor underflow	Solids contact reactor overflow	Froth skimmer underflow	Froth skimmer froth	Makeup water	Water to plant
Solids, pct	1.7	14.1	50.0	0.04	26.1	60.0	0.03	87.5	Neg	7.0	Neg	Neg	Neg	Neg	Neg
Pulp, gpm	9.1	6.1	1.6	14.5	2.2	0.7	1.5	0.3	0.4	0.9	15.5	13.9	1.6	2.9	16.8

[1] Neg, negligible.

FIGURE 3. - Flowsheet and material balance of water reclamation system.

Estimated Reagent Costs

Table 13 shows the estimated reagent cost for the selective flocculation and flotation beneficiation of the oxidized taconite. The reagent unit costs are based on bulk quantities. Reagent cost for selective flocculation and desliming was 32.7 cents/ton, flotation reagent cost was 40.9 cents/ton, the water reclamation cost was 8.7 cents/ton, and total reagent cost was 82.3 cents/ton. At a 38.5 pct weight recovery, the reagent cost per long ton of concentrate would be $2.14.

TABLE 13. - Estimated reagent cost

Reagent	Reagent cost, cents/lb[1]	Amount used, lb/ton	Cost, cents/ton of feed
SELECTIVE FLOCCULATION-DESLIMING			
Sodium hydroxide[2]	7.5	[3]2.84	21.3
Sodium silicate	4.0	1.68	6.7
Starch[4]	8.3	.56	4.7
Subtotal	NAp	NAp	32.7
FLOTATION			
Cationic collector MG-98A[5]	64.5	.56	36.1
Starch	8.3	.56	4.7
Dowfroth D-250[6]	49.0	.0014	.1
Subtotal	NAp	NAp	40.9
WATER RECLAMATION			
Lime	3.0	1.68	5.0
Superfloc 330[7]	52.2	.07	3.7
Subtotal	NAp	NAp	8.7
Total	NAp	NAp	82.3

NAp Not applicable.
[1]Reagent costs are f.o.b. quotes, April 1979, for bulk quantities.
[2]Cost per dry pound of solids in a 50-pct NaOH solution.
[3]Value includes 0.02 lb/ton used to prepare the causticized starch solutions.
[4]Causticized starch. Product of Grain Processing Corp., Muscatine, Iowa.
[5]C_8-C_{10} ether mine acetate. Product of Ashland Chemical Co., Columbus, Ohio.
[6]Propylene glycol methyl ether-type frother. Product of Dow Chemical Co., Midland, Mich.
[7]Polyamine-type reagent. Product of American Cyanamid Co., Wayne, N.J.

SUMMARY AND CONCLUSIONS

The first phase of the Bureau's research effort to insure a future domestic supply of iron raw materials for the iron and steel industry was the evaluation of a bulk sample representing an estimated 2 billion tons of oxidized taconite and containing about 35.3 pct iron. The oxidized taconite had a 1.1:1 hematite-to-goethite ratio and was beneficiated by selective flocculation and cationic flotation as an alternative method for recovering the iron oxides. Concentrates averaging 61.7 pct iron and 5.1 pct silica, with an accompanying iron recovery of 67.2 pct, were made. At a 38.5-pct weight recovery, the reagent cost per long ton of concentrate was $2.14. The metallurgical results are similar to those that can be expected in a commercial operation.

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